



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS : MacMillan, John Alexander  
FOR : PROCESS  
SERIAL NO. : 10/659,443  
FILED : September 10, 2003  
ART UNIT : 1764  
CONFIRMATION NO. : 4869  
ATTORNEY DOCKET NO. : YOUZ 2 00088

**TRANSMITTAL LETTER OF 35 U.S.C. § 119  
FOREIGN PRIORITY DOCUMENT**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

Applicant hereby claims priority under 35 U.S.C. § 119 for the above-identified U.S. patent application. This claim of priority is based upon United Kingdom application Serial No. 0302862.8, filed 07 February 2003. As required by paragraph 2 of 35 U.S.C. §119, enclosed herewith is a certified copy of GB0302862.8, filed 07 February 2003 in the United Kingdom.

Respectfully submitted,

FAY SHARPE LLP

11/9/07  
Date

  
\_\_\_\_\_  
Mark S. Svat, Reg. No. 34,261  
1100 Superior Avenue, 7<sup>th</sup> Floor  
Cleveland, OH 44114-2579  
216-861-5582

CERTIFICATE OF MAILING

I certify that this Submission of Priority Document are being deposited with the United States Postal Service as First Class mail under 37 C.F.R. § 1.8, addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on the date indicated below.

<u>11/9/07</u>	<u>George B. Sonntag</u>
Date	George B. Sonntag

Concept House  
Cardiff Road  
Newport  
South Wales  
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with patent application GB0302862.8 filed on 7 February 2003.

I also certify that the application is now proceeding in the name as identified herein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed 

Dated 29 August 2007

REGISTER ENTRY FOR GB0302862.8

Form 1 Application No GB0302862.8 filing date 07.02.2003

Title PROCESS

Applicant/Proprietor

OCTEL STARREON LLC, Incorporated in USA - Delaware, 200 Executive Drive,  
Newark, Delaware 19702, United States of America [ADP No. 07785397001]

Inventor

Classified to

C5G

C10L

Address for Service

D YOUNG & CO, 21 New Fetter Lane, LONDON, EC4A 1DA, United Kingdom  
[ADP No. 00000059006]

Application/Patent Terminated before grant 08.06.2004

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04.06.2003 Application under Section 30 filed on 02.06.2003  
Entry Type 10.1 Staff ID. JHUR Auth ID. F21

10.06.2003 OCTEL STARREON LLC, Incorporated in USA - Delaware, 200 Executive  
Drive, Newark, Delaware 19702, United States of America  
[ADP No. 07785397001]

THE ASSOCIATED OCTEL COMPANY LIMITED, Incorporated in the United  
Kingdom, Global House, Bailey Lane, MANCHESTER, M90 4AA, United  
Kingdom [ADP No. 07439292003]

registered as Applicant/Proprietor in place of  
OCTEL STARREON LLC, Incorporated in USA - Delaware, 200 Executive  
Drive, Newark, Delaware 19702, United States of America  
[ADP No. 07785397001]

by virtue of assignment dated 24.03.2003. Form 21/77 and documents  
filed on GB0302862.8.

Entry Type 8.4 Staff ID. CST1 Auth ID. F21

21.09.2004 Notification of change of Address For Service address of  
D YOUNG & CO, 21 New Fetter Lane, LONDON, EC4A 1DA, United Kingdom  
[ADP No. 00000059006]

to  
D YOUNG & CO, 120 Holborn, LONDON, EC1N 2DY, United Kingdom  
[ADP No. 00000059006]  
dated 20.09.2004. Written notification filed on GB2393339  
Entry Type 7.3 Staff ID. LDAV Auth ID. F20

29.08.2007 Notification of change of Address For Service name and address of  
D YOUNG & CO, 120 Holborn, LONDON, EC1N 2DY, United Kingdom  
[ADP No. 00000059006]

to  
APPLEYARD LEES, 15 Clare Road, HALIFAX, West Yorkshire, HX1 2HY,  
United Kingdom [ADP No. 00000190001]  
dated 14.08.2007. Written notification filed on GB0302862.8  
Entry Type 7.1 Staff ID. RCOX Auth ID. A3

TIMED: 29/08/07 11:53:16

PAGE: 2

REGISTER ENTRY FOR GB0302862.8 (Cont.)

\*\*\*\*\* END OF REGISTER ENTRY \*\*\*\*\*

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OPTICS. - PATENTS

29/08/07 11:53:28  
PAGE: 1

RENEWAL DETAILS

APPLICATION NUMBER GB03028628

PROPRIETOR(S)

Octel Starreon LLC, Incorporated in USA - Delaware, 200 Executive Drive, Newark, Delaware 19702, United States of America

The Associated Octel Company Limited, Incorporated in the United Kingdom, Global House, Bailey Lane, MANCHESTER, M90 4AA, United Kingdom

DATE FILED 07.02.2003

DATE GRANTED

DATE NEXT RENEWAL DUE

DATE NOT IN FORCE 08.06.2004

DATE OF LAST RENEWAL

YEAR OF LAST RENEWAL 00

STATUS TERMINATED BEFORE GRANT

\*\*\*\*\* END OF REPORT \*\*\*\*\*



GB 0302862.8

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of

OCTEL STARREON LLC,  
200 Executive Drive,  
Newark,  
Delaware 19702,  
United States of America

Incorporated in USA - Delaware,

[ADP No. 07785397001]

and

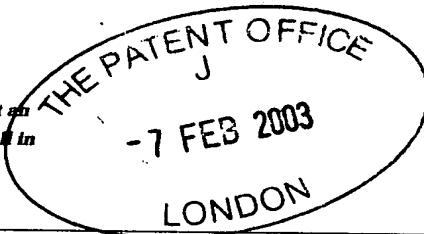
THE ASSOCIATED OCTEL COMPANY LIMITED,  
Incorporated in the United Kingdom,  
Global House,  
Bailey Lane,  
MANCHESTER,  
M90 4AA,  
United Kingdom

[ADP No. 07439292003]

10FEB03 E783399-1 D02246  
P01/7700 0.00-0302862.8

## Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road  
Newport  
South Wales  
NP10 8QQ

## 1. Your reference

P012310GB DAA

## 2. Patent application number

(The Patent Office will fill in this part)

07 FEB 2003

0302862.8

## 3. Full name, address and postcode of the or of each applicant (underline all surnames)

0718 5397001

Octel Starreon LLC  
200 Executive Drive  
Newark, DE 19702  
United States of America

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

United States of America

SECTION 30 (1977 ACT) APPLICATION FILED 2/6/03

Delaware Al 21/2/03

## 4. Title of the invention

Process

## 5. Name of your agent (if you have one)

D Young &amp; Co

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

D Young & Co  
120 Holborn  
LONDON  
EC1N 2DY

Patents ADP number (if you know it)

59006



## 6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)Date of filing  
(day / month / year)

## 7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
(day / month / year)

## 8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

Yes

- a) any applicant named in part 3 is not an inventor, or
- b) there is an inventor who is not named as an applicant, or
- c) any named applicant is a corporate body.

See note (d))

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form.  
Do not count copies of the same document

Continuation sheets of this form 0

Description 16

 Claim(s) 4

Abstract 1

Drawing(s) 0

10. If you are also filing any of the following, state how many against each item.

Priority documents 0

Translations of priority documents 0

Statement of inventorship and right 0  
to grant of a patent (Patents Form 7/77)

Request for preliminary examination Yes  
and search (Patents Form 9/77)

Request for substantive examination 0  
(Patents Form 10/77)

Any other documents 0  
(please specify)

I/We request the grant of a patent on the basis of this application.

11.

Signature

 D Young & Co (Agents for the Applicants)

Date 7 February 2003

12. Name and daytime telephone number of person to contact in the United Kingdom

David Alcock

023 8071 9500

**Warning**

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

**Notes**

- a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 08459 500505.
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PROCESS

The present invention relates to a process. In particular the present invention relates to a process for the production of a fuel additive and a fuel composition.

5

It is well known to those skilled in the art that hydrocarbon liquids such as fuels may corrode the metal surfaces with which they come in contact. In order to address these corrosion problems, corrosion inhibitors are often added to fuels in order to reduce or prevent corrosion of the systems in which the fuels are stored and/or handled.

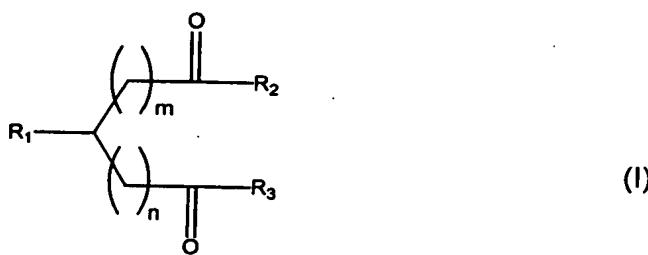
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In certain oil refinery applications a corrosion inhibitor is required which will be resistant to base neutralisation. The base, typically NaOH, can be present in fuels that have undergone a refinery sweetening treatment or acid neutralisation. Normally, but not exclusively, the corrosion inhibitor is added after "caustic wash". Furthermore, during 15 distribution a fuel may come in contact with associated caustic water bottoms so the corrosion inhibitor deactivation may occur within the distribution system. The consequence of base neutralisation is corrosion inhibitor deactivation, precipitate formation and consequent levels of rust which are typical of a fuel without added corrosion inhibitor.

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The present invention alleviates the problems of the prior art.

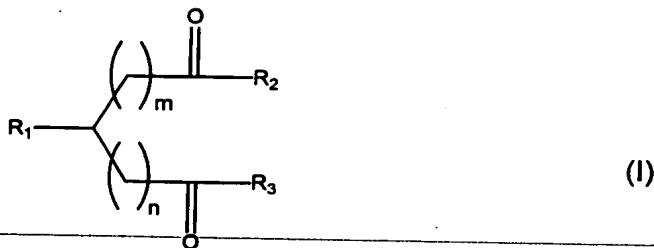
In one aspect the present invention provides a process for the production of a fuel composition having a NACE corrosion rating of between 0% and 25%, comprising the 25 steps of (i) contacting a fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least 30 one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; and (ii)

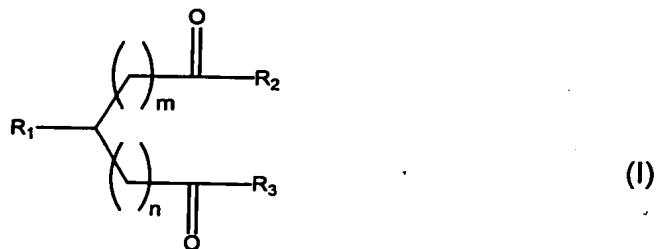
contacting the initial fuel composition with a caustic material to provide the fuel composition without subsequent addition of a corrosion inhibitor.

In one aspect the present invention provides a process for the production of a fuel composition suitable for final use, comprising the steps of (i) contacting a fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; and (ii) contacting the initial fuel composition with a caustic material to provide the fuel composition without subsequent addition of a corrosion inhibitor.

In one aspect the present invention provides a process for the production of a fuel composition comprising the steps of (i) contacting a fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition

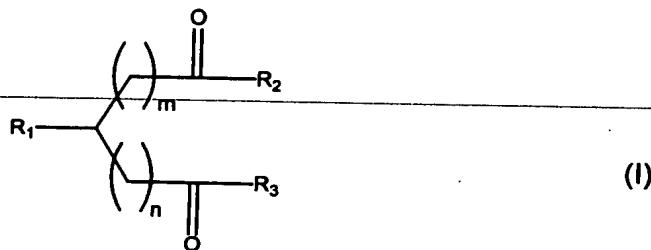


wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; and (ii) contacting the initial fuel composition with a caustic material to provide the fuel composition without subsequent addition of a corrosion inhibitor; wherein at least 10%, preferably at least 20%, more preferably at least 40%, more preferably at least 60%,

more preferably at least 80% of the corrosion inhibitor of formula (I) present and active in the initial fuel composition is present and active in the fuel composition.

5 In one aspect the present invention provides a fuel composition obtained or obtainable by a process as herein defined.

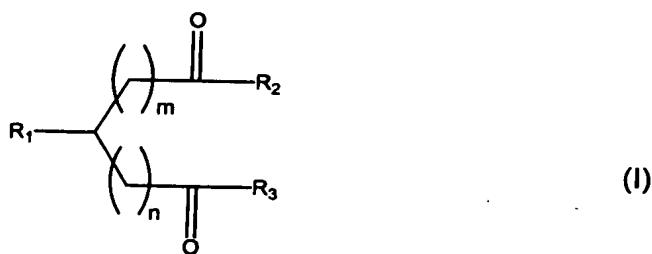
In one aspect the present invention provides a method of inhibiting corrosion on a metal surface exposed to a fuel comprising the steps of (i) contacting the fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



10 wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; (ii) contacting the initial fuel composition with a caustic material to provide a fuel composition; and (iii) exposing the metal surface to the fuel composition.

15

In one aspect the present invention provides use of a corrosion inhibitor of formula (I) for providing caustic wash resistant corrosion inhibition



wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an 20 optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—.

It has surprisingly been found that corrosion inhibitors of formula (I) typically retain their corrosion inhibiting properties when contacted with a caustic material. In the prior art, many corrosion inhibitors used in fuel were significantly deactivated by contact with a caustic material. The result was that fuel treated with such corrosion inhibitors displayed

5 levels of corrosion following either a caustic wash or other contact with a caustic material that were typical of untreated fuel. This frequently necessitated the subsequent addition of further corrosion inhibitor in order for the fuel to satisfy industry standards relating to corrosion. Commonly, the deactivated corrosion inhibitor problematically precipitates from the fuel, potentially causing blocked filters. In contrast, fuels treated with a

10 corrosion inhibitor of formula (I) display acceptable anti-corrosion characteristics even after contact with a caustic material. Thus, when a corrosion inhibitor of formula (I) is dosed into a fuel, addition of further corrosion inhibitor following a caustic wash or other contact with a caustic material may typically be avoided. Eliminating this final re-addition step provides numerous benefits including reduced cost, improved fuel quality and

15 improved manufacturing logistics.

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It is well known that the amount of sulphur contained in a fuel will decrease . For example, in anticipation of the US EPA 2006 ULSD Regulations, it is expected that sulphur levels will be progressively decreasing. The level of sulphur in fuel additives

20 treated at the terminal will be limited to 15 ppm. Many of the corrosion inhibitors currently in use contain sulphur. Corrosion inhibitors of formula (I) typically contain no sulphur and thus provide a further advantage over many other corrosion inhibitors.

It has surprisingly been found that corrosion inhibitors of formula (I) also increase the

25 lubricity of a fuel to which they are added. Increased lubricity prevents wear on contacting metal surfaces. The amount of wear to a surface may be measured for example by well-known tests such as the wear scar test. Corrosion inhibitors of formula (I) may therefore be used as multi-functional additives acting both as corrosion inhibitors and as lubricity additives. Therefore a fuel composition comprising a corrosion inhibitor

30 of formula (I) may advantageously not comprise any additional lubricity additive.

The term "NACE corrosion rating" as used herein means the percentage corrosion obtained according to the NACE Standard Test Method for determining the corrosive properties of cargoes in petroleum product pipelines (TM0172-2001). Further

35 information about this NACE Standard Test Method may be obtained from NACE

International, 1440 South Creek Drive, Houston or from the NACE International website [www.http://nace.org](http://nace.org)

The term "fuel" as used herein refers to any liquid hydrocarbon fuel. Typical examples of  
5 a liquid hydrocarbon fuels are gasoline and diesel. As used herein, "gasoline" refers to  
motor fuels meeting ASTM standard D439 and "diesel" refers to middle distillate fuels  
meeting ASTM standard D975, and includes blends of hydrocarbon fuels with  
oxygenated components, such as MTBE, ETBE, ethanol, etc. as well as the distillate  
fuels themselves. The fuels may be leaded or unleaded and may contain, in addition to  
10 the additive compositions of this invention, any of the other additives conventionally  
added to gasoline, such as scavengers, anti-icing additives, octane requirement  
improvers, detergent packages, antioxidants, demulsifiers, corrosion inhibitors etc.

The term "hydrocarbyl" as used herein refers to a group comprising at least C and H that  
15 may optionally comprise one or more other suitable substituents. Examples of such  
substituents may include halo-, alkoxy-, nitro-, an alkyl group, or a cyclic group. In  
addition to the possibility of the substituents being a cyclic group, a combination of  
substituents may form a cyclic group. If the hydrocarbyl group comprises more than one  
20 C then those carbons need not necessarily be linked to each other. For example, at  
least two of the carbons may be linked *via* a suitable element or group. Thus, the  
hydrocarbyl group may contain heteroatoms. Suitable heteroatoms will be apparent to  
those skilled in the art and include, for instance, sulphur, nitrogen, oxygen, silicon and  
phosphorus.

25 The term "hydrocarbyl-OH" refers to a hydrocarbyl group with a terminal hydroxy  
substituent.

A typical hydrocarbyl group is a hydrocarbon group. Here the term "hydrocarbon" means  
any one of an alkyl group, an alkenyl group, an alkynyl group, which groups may be  
30 linear, branched or cyclic, or an aryl group. The term hydrocarbon also includes those  
groups but wherein they have been optionally substituted. If the hydrocarbon is a  
branched structure having substituent(s) thereon, then the substitution may be on either  
the hydrocarbon backbone or on the branch; alternatively the substitutions may be on the  
hydrocarbon backbone and on the branch.

The term "caustic material" as used herein relates to a material comprising at least one metal hydroxide or alkaline material. The term "alkaline material" means a material with a pH of greater than 7 when in aqueous solution.

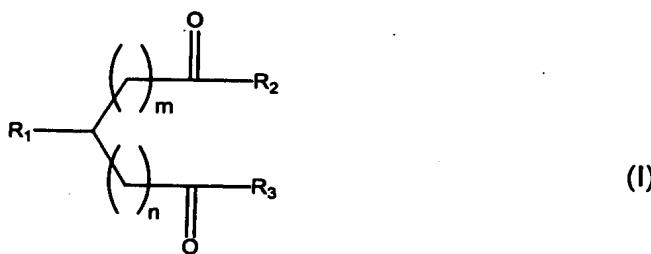
- 5 The term "a fuel composition suitable for final use" as used herein relates to a finished fuel composition complying with industry standards relating to corrosion. It will be appreciated that the term "finished" means in a suitable condition to leave the refinery having met the approved regulatory standards.
- 10 The term "metal surface" relates to any surface comprising at least one metal. The metal surface typically comprises iron and may for example comprise an iron-containing alloy such as carbon steel. The metal surface is typically a pipeline or other metal vessel used in fuel transport and/or refinery processes.
- 15 The term "caustic wash" as used herein means contacting a fluid with an alkaline solution.

The term "caustic wash resistant corrosion inhibition" as used herein means the level of corrosion inhibition following a caustic wash is not more than 25% lower than the level of

- 20 corrosion inhibition prior to the caustic wash. The corrosion inhibition is preferably measured using the NACE Standard Test Method TM0172-2001. Typically, a corrosion inhibitor which provides caustic wash resistant corrosion inhibition will achieve a NACE corrosion rating in a fuel less than 5% corrosion prior to a caustic wash. 0% corrosion indicates 100% corrosion inhibition. Following a caustic wash, the same corrosion
- 25 inhibitor will achieve a NACE corrosion rating in the fuel of not more than 25% corrosion. 25% corrosion indicates 75% corrosion inhibition. Thus in the typical case there is a reduction in corrosion inhibition from 100% to not less than 75%. In other words, there is a reduction in corrosion inhibition of not more than 25%.

- 30 **Corrosion Inhibitor of Formula (I)**

As previously mentioned, in one aspect the present invention provides a process for the production of a fuel composition having a NACE corrosion rating of between 0% and 25%, comprising the steps of (i) contacting a fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; and (ii)

5 contacting the initial fuel composition with a caustic material to provide the fuel composition without subsequent addition of a corrosion inhibitor.

m and n

10 Preferably m and n are each independently an integer from 0 to 9, preferably 0 to 8, preferably 0 to 7, preferably 0 to 6, more preferably 0 to 5.

Preferably m and n are each independently an integer selected from 0, 1, 2 and 3.

15 In one aspect, preferably one of m and n is 0. In this aspect, preferably the other of m and n is other than 0.

Preferably in one aspect, one of m and n is 0 and the other of m and n is 1.

20 R<sub>1</sub>

As previously mentioned, the corrosion inhibitor of formula (I) comprises the group R<sub>1</sub>, wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group.

25 In one aspect, R<sub>1</sub> is an optionally substituted hydrocarbon group.

As previously mentioned, the term "hydrocarbon" as used herein means any one of an alkyl group, an alkenyl group, an alkynyl group, which groups may be linear, branched or cyclic, or an aryl group. The term hydrocarbon also includes those groups but wherein

30 they have been optionally substituted. If the hydrocarbon is a branched structure having

substituent(s) thereon, then the substitution may be on either the hydrocarbon backbone or on the branch; alternatively the substitutions may be on the hydrocarbon backbone and on the branch.

5 Preferably  $R_1$  is an optionally substituted alkyl or alkenyl group. In one aspect  $R_1$  is an optionally substituted alkyl group. In another aspect,  $R_1$  is an optionally substituted alkenyl group.

10 The term "alkenyl" refers to a branched or straight chain hydrocarbon, which can comprise one or more carbon-carbon double bonds. Exemplary alkenyl groups include propylenyl, butenyl, isobutenyl, pentenyl, 2,2-methylbutenyl, 3-methylbutenyl, hexanyl, heptenyl, octenyl, and polymers thereof.

15 In one aspect  $R_1$  is an optionally substituted branched alkyl or alkenyl group. Preferably,  $R_1$  is a polyisobut enyl (PIB) group.

20 Conventional PIBs and so-called "high-reactivity" PIBs (see for example EP-B-0565285) are suitable for use in this invention. High reactivity in this context is defined as a PIB wherein at least 50%, preferably 70% or more, of the terminal olefinic double bonds are of the vinylidene type, for example the GLISSOPAL compounds available from BASF.

25 In one aspect  $R_1$  has between 5 and 200 carbon atoms, preferably between 10 and 200 carbon atoms, preferably between 10 and 100 carbon atoms, preferably between 10 and 40 carbon atoms, preferably between 12 and 32 carbon atoms such as between 12 and 26 carbon atoms.

30 In one aspect,  $R_1$  has a molecular weight of from 100 to 2000, preferably from 200 to 800, preferably from 200 to 500, more preferably from 250 to 400 such as 260 or 360.

35  $R_2$ ,  $R_3$ ,  $R_4$  and  $R_5$

As previously mentioned, the corrosion inhibitor of formula (I) comprises the groups  $R_2$  and  $R_3$ , wherein either  $R_2$  is  $OR_4$  and  $R_3$  is  $OR_5$ , wherein  $R_4$  and  $R_5$  are selected from hydrogen and hydrocarbyl-OH and wherein at least one of  $R_4$  and  $R_5$  is hydrogen; or  $R_2$  and  $R_3$  together represent  $—O—$ .

In a preferred aspect,  $R_2$  is  $OR_4$  and  $R_3$  is  $OR_5$ .

In one embodiment preferably one of  $R_4$  and  $R_5$  is hydrogen and the other of  $R_4$  and  $R_5$  is hydrocarbyl-OH.

5

Preferably  $R_4$  and  $R_5$  are selected from hydrogen and  $(C_xH_{2x})-OH$  wherein  $x$  is an integer of at least 1. Preferably  $x$  is an integer from 1 to 30, preferably 1 to 20, more preferably 1 to 10. In one aspect, one of  $R_4$  and  $R_5$  is hydrogen and the other of  $R_4$  and  $R_5$  is  $(C_xH_{2x})-OH$ .

10

More preferably,  $R_4$  and  $R_5$  are selected from hydrogen and  $(CH_2)_y-OH$  wherein  $y$  is an integer of at least 1. Preferably  $y$  is an integer from 1 to 30, preferably 1 to 20, more preferably 1 to 10. In one aspect, one of  $R_4$  and  $R_5$  is hydrogen and the other of  $R_4$  and  $R_5$  is  $(CH_2)_y-OH$ .

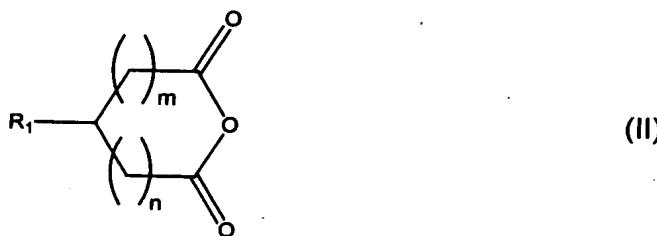
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In a preferred embodiment each of  $R_4$  and  $R_5$  is hydrogen.

In one highly preferred embodiment, in the corrosion inhibitor of formula (I), one of  $m$  and  $n$  is 0 and the other of  $m$  and  $n$  is 1,  $R_1$  is a polyisobutetyl group with a molecular weight 20 of approximately 260,  $R_2$  is  $OR_4$ ,  $R_3$  is  $OR_5$  and each of  $R_4$  and  $R_5$  is hydrogen.

25

In one highly preferred embodiment, in the corrosion inhibitor of formula (I), one of  $m$  and  $n$  is 0 and the other of  $m$  and  $n$  is 1,  $R_1$  is a polyisobutetyl group with a molecular weight of approximately 260 or 360,  $R_2$  is  $OR_4$ ,  $R_3$  is  $OR_5$  and each of  $R_4$  and  $R_5$  is hydrogen.



Preferred Quantities

In one aspect, in step (i), the fuel is treated with 0.25 to 20 ptb of a corrosion inhibitor of formula (I), preferably 1 to 15 ptb, preferably 1 to 12 ptb, more preferably 1 to 10 ptb.

5

Ptb is an abbreviation for pounds per thousand barrels. 1 ptb is equivalent to 2.85mg/L.

In one preferred aspect, in step (i), the fuel is treated with 1 to 5 ptb of a corrosion inhibitor of formula (I), preferably 1, 2 or 3 ptb.

10

Caustic Material

As previously mentioned, step (ii) of the process of the present invention involves contacting the initial fuel composition with a caustic material to provide the fuel 15 composition without subsequent addition of a corrosion inhibitor.

Preferably the caustic material is an alkaline solution. The term "alkaline solution" as used herein refers to an aqueous solution with a pH of greater than 7. In one aspect the caustic material is a 0.001% to 30% w/w alkaline solution, such as a 1% to 10% w/w 20 alkaline solution, such as a 3% w/w alkaline solution, a 4% w/w alkaline solution or a 5% w/w alkaline solution.

In one aspect, the caustic material comprises a water-soluble metal hydroxide. Preferably the caustic material comprises a hydroxide of a metal from group 1 or group 2 25 of the periodic table. In one preferred aspect, the caustic material is an aqueous solution of sodium hydroxide ( $\text{NaOH}_{(\text{aq})}$ ) or an aqueous solution of potassium hydroxide ( $\text{KOH}_{(\text{aq})}$ ). Preferably, the caustic material is an aqueous solution of sodium hydroxide ( $\text{NaOH}_{(\text{aq})}$ ).

NACE Corrosion Rating

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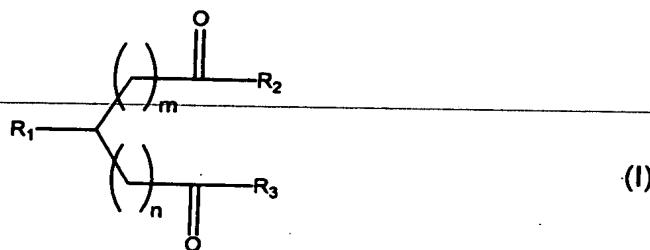
As previously mentioned, the present invention relates to a process for the production of a fuel composition having a NACE corrosion rating of between 0% and 25%.

In a preferred aspect, the fuel composition has a NACE corrosion rating of between 0% 35 and 20%, preferably between 0% and 15%, preferably between 0% and 10%, more

preferably between 0% and 5%. In a highly preferred aspect the fuel composition has a NACE corrosion rating of between 0% and 1%, such as between 0% and 0.5% or between 0% and 0.1%.

5 **Method**

In one aspect the present invention provides a method of inhibiting corrosion on a metal surface exposed to a fuel comprising the steps of (i) contacting the fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



10. wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; (ii) contacting the initial fuel composition with a caustic material to provide a fuel composition; and (iii) exposing the metal surface to the fuel composition.

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In this aspect, preferably the corrosion inhibitor of formula (I) is as herein defined. In this aspect, preferably step (i) is as herein defined. In this aspect, preferably step (ii) is as herein defined.

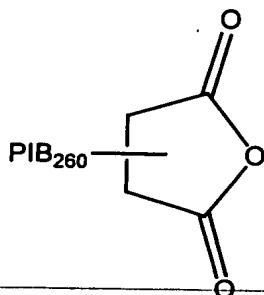
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In this aspect, preferably the corrosion inhibitor of formula (I) is as herein defined and/or step (i) is as herein defined and/or step (ii) is as herein defined.

Aspects of the invention are defined in the appended claims.

25

The present invention will now be described in further detail in the following examples.

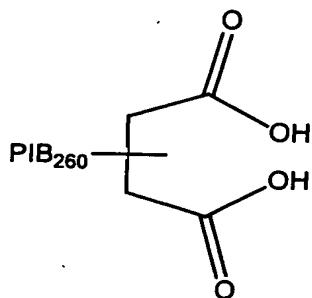
**EXAMPLES****Syntheses****PIBSA (Polyisobutetyl Succinic Anhydride)**

PIBSA 260

5 260 mwt high reactive polyisobutene (PIB) (642.3g) was stirred in a 1l oil jacketed reactor equipped with an overhead stirrer and thermometer. The PIB was heated to 200°C under a nitrogen atmosphere. Maleic anhydride (0.65 mole equivalents, 157.46g) was charged to the reactor over a 3-hour period, whilst maintaining 195°C to 200°C. The reaction mixture was then heated to 205°C for an 8-hour period. Whilst at 205°C, a

10 vacuum was slowly pulled on the reactor for a 1.5 hour period to remove excess maleic anhydride to <0.1% m/m. 768.9g of product was isolated. Analysis of product gave a maleic anhydride content <0.1% m/m, a PIB content of 37 %m/m and an Acid Value of 5.26 mmolH+/g.

15 PIBSA 360 may be made by the same method using 360 mwt PIB in place of 260 mwt PIB.

**PIBS Acid (Polyisobutetyl Succinic Acid) - a corrosion inhibitor of formula (I)**

PIBS Acid 260

20 260 mwt high reactive PIB-derived PIBSA (667.5g) was stirred with xylene (40% m/m, 445.0g) at room temperature, in a 1l oil jacketed reactor equipped with an overhead

stirrer, thermometer and condenser. Whilst at room temperature, the water (0.9 mole equivalents, 28.44g) was charged whilst stirring and the reaction mixture heated to 90°C for 3 hours. The solvent content and conversion was confirmed by analytical. 845.57g of product was isolated.

5

Analysis of product gave a solvent content of 39 %m/m and PIBSA content of 2% m/m.

PIBS Acid 360 may be made by the same method using 360 mwt PIB in place of 260 mwt PIB.

10

#### NACE Rust Test (TM 0172)

A standardised corrosion test, such as the National Association of Corrosion Engineers (NACE) standard test TM-01-72, can measure the effectiveness of corrosion inhibitors 15 which are introduced into pipeline cargoes to prevent rusting caused by traces of moisture condensing from the products. The results of such a test are reported as a relative rating on the scale A-E.

Rating	Percentage Corrosion
A	None
B++	Less than 0.1% (2 or 3 spots of no more than 1mm diameter)
B+	Less than 5%
B	5% to 25%
C	25% to 50%
D	50% to 75%
E	75% to 100%

20 Table 1 - NACE Rust Test (TM 0172) Before Caustic Washing

Fuel	PIBSA 360, ptb	Rating/% Corrosion
Isopar M	2	A/0
Isooctane	2	A/0

PIBSA 360 = Polyisobutylene Succinic Anhydride (PIB Mwt.360)

Fuel	DCI-30 ptb	Rating/% Corrosion
Isopar M	2	A/0
Isooctane	2	A/0
Gasoline	2	A/0
Diesel	2	A/0

DCI-30 is 63% PIBS Acid 260 (Polyisobutylene Succinic Acid (PIB Mwt. 260)) and 37% xylene.

5

The above work has been performed

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These results demonstrate that DCI-30 provides excellent corrosion inhibition in different fuels.

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Table 2 - NACE Rust Test (TM 0172) After Caustic Washing

15 A sample of Canadian gasoline was dosed with varying amounts of a composition of 63% PIBS Acid 260 and 37% xylene. This composition is referred to as DCI-30 in the table below. In order to provide an aggressive test of the additive's resistance to caustic disarming, the gasoline sample was then washed with 5% vol/vol of a 4% NaOH solution according to the following method:

1. Make a 4% NaOH solution in deionized water.
- 20 2. Pour 400 ml of the gasoline sample into a 500 mL separatory funnel. Add 40 ml of the 4% NaOH solution.
3. Shake vigorously for 5 minutes, venting occasionally.
4. Allow layers to separate - about 30 minutes.
5. Drain off aqueous layer.
- 25 6. Perform NACE rust test on the gasoline sample.

30 The following results were obtained. Results for traditional dimer acid based corrosion inhibitor chemistries, Trad A and Trad B, are included for purposes of comparison. Trad B is a traditional dimer acid corrosion inhibitor based on conventional tall oil fatty acid chemistry. Trad A is a traditional corrosion inhibitor based on conventional tall oil fatty acid chemistry in combination with a synthetic synergist.

Fuel	DCI-30 ptb	Rating/% Corrosion
Canadian RUL Gasoline	0	E/90
Canadian RUL Gasoline	3	B/15
Canadian RUL Gasoline	4	A/0

\* The NACE rating of untreated gasoline is E99

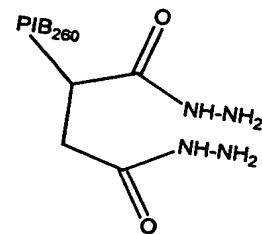
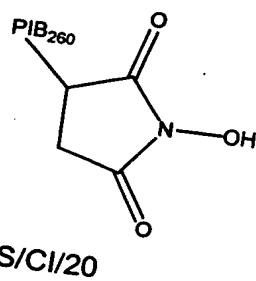
Fuel	Corrosion Inhibitor	ptb	Rating/% Corrosion (unwashed)	Rating/% Corrosion (washed)
Isooctane	-	-	D 65%	E 99%
Isooctane	DCI-30	2	A 0%	B+ 3%
Isooctane	DCI-30	5	A 0%	A 0%
Isooctane	Trad A	2	A 0%	E 80%
Isooctane	Trad A	5	A 0%	E 80%
Isooctane	Trad B	5	A 0%	E 85%
Isopar M	-	-	E 85%	D 60%
Isopar M	DCI-30	2	A 0%	A 0%
Isopar M	DCI-30	5	A 0%	B++ <0.1%
Isopar M	Trad A	2	A 0%	E 95%
Isopar M	Trad A	5	A 0%	E 99%
Isopar M	Trad B	5	A 0%	E 99%
2002 RUL Gasoline	-	-	E 90%	D 60%
2002 RUL Gasoline	DCI-30	2	A 0%	C 40%
2002 RUL Gasoline	DCI-30	5	A 0%	B 20%
2002 RUL Gasoline	Trad A	2	A 0%	D 60%
2002 RUL Gasoline	Trad A	5	A 0%	C 50%
2002 RUL Gasoline	Trad B	5	B+ 5%	E 85%
Diesel	-	-	E 80%	D 70%
Diesel	DCI-30	2	A 0%	C 50%
Diesel	DCI-30	5	A 0%	B 15%
Diesel	Trad A	2	A 0%	E 85%
Diesel	Trad A	5	A 0%	E 98%
Diesel	Trad B	5	A 0%	E 95%

5 These results demonstrate that by use of a corrosion inhibitor of formula (I) such as PIBS Acid 260, good corrosion inhibition is retained following a caustic wash.

Table 3 – NACE Rust Test (TM 0172) Comparison with other Corrosion Inhibitors

ptb	Isopar M				NACE Rating Isooctane				Gasoline washed with 4% NaOH solution	
	0	1	2	3	0	1	2	3	0	2
PIBS Acid 260	E85	B++<0.1	A0	A0	E95	B++<0.1	A0	A0	E80	B25
KS/Cl/20	E85	C40	C40	B+1	E95	B++<0.1	A0	A0	E80	B10
KS/Cl/21	E85	E85	D75	B20	E95	C40	A0	A0	E80	C40
Trad B	E85	B++<0.1	A0	A0	E95	B++<0.1	A0	A0	E80	E90

\* The NACE rating of untreated gasoline is E99.



These results demonstrate that PIBS Acid 260 provides good corrosion inhibition compared with other additives. Following a caustic wash the level of corrosion inhibition provided by PIBS Acid 260 remains high.

All publications mentioned in the above specification are herein incorporated by reference. Various modifications and variations of the described methods and system of the invention will be apparent to those skilled in the art without departing from the scope and spirit of the invention. Although the invention has been described in connection with specific preferred embodiments, it should be understood that the invention as claimed should not be unduly limited to such specific embodiments. Indeed, various modifications of the described modes for carrying out the invention which are obvious to those skilled in chemistry or related fields are intended to be within the scope of the following claims.

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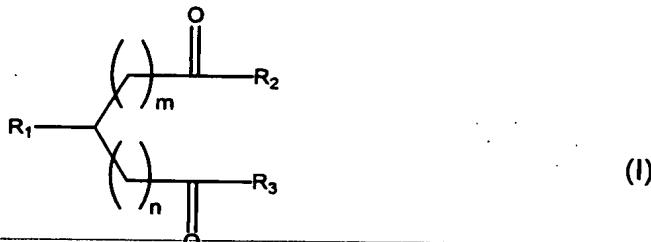
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**CLAIMS**

1. A process for the production of a fuel composition having a NACE corrosion rating of between 0% and 25%, comprising the steps of:
  - 5 (i) contacting a fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



wherein m and n are each independently an integer from 0 to 10;

wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group;

wherein

- 10 10 either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—; and
- 15 (ii) contacting the initial fuel composition with a caustic material to provide the fuel composition without subsequent addition of a corrosion inhibitor.

2. A process according to claim 1 wherein m and n are each independently an integer from 0 to 5.

- 20 3. A process according to claim 1 or 2 wherein one of m and n is 0 and the other of m and n is 1.

4. A process according to claim 1, 2 or 3 wherein R<sub>1</sub> is an optionally substituted hydrocarbon group.

25

5. A process according to any one of the preceding claims wherein R<sub>1</sub> is an optionally substituted alkyl or alkenyl group.

- 30 6. A process according to any one of the preceding claims wherein R<sub>1</sub> is an optionally substituted branched alkyl or alkenyl group.

7. A process according to any one of the preceding claims wherein  $R_1$  is a polyisobutetyl group.
- 5 8. A process according to any one of the preceding claims wherein  $R_1$  has between 10 and 200 carbon atoms.
9. A process according to any one of the preceding claims wherein  $R_1$  has between 12 and 32 carbon atoms.
- 10 10. A process according to any one of the preceding claims wherein  $R_1$  has a molecular weight of from 250 to 400.
11. A process according to any one of the preceding claims wherein  $R_1$  has a molecular weight of approximately 260 or approximately 360.
12. A process according to any one of the preceding claims wherein  $R_2$  is  $OR_4$  and  $R_3$  is  $OR_5$ .
- 20 13. A process according to any one of the preceding claims wherein  $R_4$  and  $R_5$  are selected from hydrogen and  $(C_xH_{2x})-OH$  wherein x is an integer of at least 1.
14. A process according to any one of the preceding claims wherein  $R_4$  and  $R_5$  are selected from hydrogen and  $(CH_2)_y-OH$  wherein y is an integer of at least 1.
- 25 15. A process according to any one of the preceding claims wherein  $R_4$  and  $R_5$  are both hydrogen.
- 30 16. A process according to any one of the preceding claims wherein one of m and n is 0 and the other of m and n is 1,  $R_1$  is a polyisobutetyl group with a molecular weight of approximately 260 or 360,  $R_2$  is  $OR_4$ ,  $R_3$  is  $OR_5$  and  $R_4$  and  $R_5$  are both hydrogen.
17. A process according to any one of the preceding claims wherein, in step (i), the fuel is treated with 1 to 20 ptb of a corrosion inhibitor of formula (I).

18. A process according to any one of the preceding claims wherein, in step (i), the fuel is treated with 1 to 10 ptb of a corrosion inhibitor of formula (I).

19. A process according to any one of the preceding claims wherein, in step (ii), the  
5 caustic material is an alkaline solution.

20. A process according to any one of the preceding claims wherein, in step (ii), the caustic material is a 0.001% - 30% w/w alkaline solution.

10 21. A process according to any one of the preceding claims wherein, in step (ii), the caustic material is a 1% - 10% w/w alkaline solution.

22. A process according to any one of the preceding claims wherein, in step (ii), the caustic material is NaOH(aq) or KOH(aq).

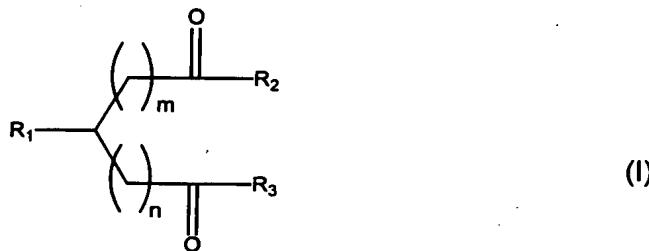
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23. A process according to any one of the preceding claims wherein, in step (ii), the caustic material is NaOH(aq).

20 24. A fuel composition obtained or obtainable by a process according to any one of the preceding claims.

25. A method of inhibiting corrosion on a metal surface exposed to a fuel comprising the steps of:

(i) contacting the fuel with a corrosion inhibitor of formula (I) to provide an initial fuel  
composition



wherein m and n are each independently an integer from 0 to 10;

wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group;

wherein

either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and

30 hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen;

or  $R_2$  and  $R_3$  together represent —O—;

(ii) contacting the initial fuel composition with a caustic material to provide a fuel composition; and

(iii) exposing the metal surface to the fuel composition.

5

26. A method according to claim 25 wherein the corrosion inhibitor of formula (I) is as defined in any one of claims 2 to 16 and/or step (i) is as defined in either of claims 17 or 18 and/or step (ii) is as defined in any one of claims 19 to 23.

10 27. Use of a corrosion inhibitor of formula (I) as defined in any one of claims 1 to 16 for providing caustic wash resistant corrosion inhibition.

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28. A process substantially as hereinbefore described with reference to the Examples.

15 29. A fuel composition substantially as hereinbefore described with reference to the Examples.

30. A method substantially as hereinbefore described with reference to the Examples.

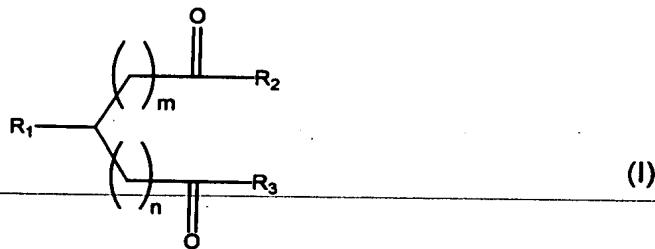
20 31. Use substantially as hereinbefore described with reference to the Examples.

ABSTRACT

## PROCESS

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The present invention provides a process for the production of a fuel composition having a NACE corrosion rating of between 0% and 25%, comprising the steps of: (i) contacting a fuel with a corrosion inhibitor of formula (I) to provide an initial fuel composition



10 wherein m and n are each independently an integer from 0 to 10; wherein R<sub>1</sub> is an optionally substituted hydrocarbyl group; wherein either R<sub>2</sub> is OR<sub>4</sub> and R<sub>3</sub> is OR<sub>5</sub>, wherein R<sub>4</sub> and R<sub>5</sub> are selected from hydrogen and hydrocarbyl-OH and wherein at least one of R<sub>4</sub> and R<sub>5</sub> is hydrogen; or R<sub>2</sub> and R<sub>3</sub> together represent —O—, and (ii)

15 contacting the initial fuel composition with a caustic material to provide the fuel composition without subsequent addition of a corrosion inhibitor.